Characterization of the Various Sediments Collected for Toxicity Testing in Support of the *Deepwater Horizon*Natural Resource Damage Assessment Technical Report

Prepared for:

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1. Introduction

The Trustees' *Deepwater Horizon* (DWH) natural resource damage assessment (NRDA) toxicity testing program included many bioassays of sediments contaminated with DWH oil. The exposure methods that we used during these bioassays varied with the species and lifestage of interest (Morris et al., 2015). During the sediment toxicity testing program, we conducted bioassays using various preparations of contaminated sediment: exposure to sediments collected from areas that were contaminated with DWH oil during the spill (Section 3.1), exposure to sediment that we spiked with an oil:artificial saltwater emulsion (Section 3.2), and exposure to reference sediments that we spiked with DWH oil in the laboratory (Section 3.3). Below, we describe the differences in the physical and chemical properties of the sediment types used during the NRDA and discuss the rationale behind using different preparations of contaminated sediments.

2. General Information

There were several efforts to collect sediments for the purpose of toxicity testing. The Trustees opportunistically collected contaminated sediments from the field during the DWH response effort. During subsequent years, they returned to collect sediments from the same locations and also collected sediments from additional reference (uncontaminated) locations. For the remainder of this report we refer to these sediments as being "collected during the response." We conducted bioassays using these sediments for the NRDA. Additionally, as a part of the NRDA toxicity program, we collected reference sediments that we could apply oil to for use in bioassays. For the remainder of this report we refer to these sediments as being "collected for spiking." Before using any of these samples for toxicity testing, we sent them to an analytical laboratory where they were screened for several different types of contamination. We did not include any samples in our toxicity testing that had elevated levels of contaminants, other than the contaminants of interest – polycyclic aromatic hydrocarbons (PAHs).

2.1 Sediment Collection

2.1.1 Sediments collected during the response

During the response effort the Trustees opportunistically collected contaminated sediment samples. During subsequent years, the Trustees collected additional sediment samples from the sites that were visited during the response effort and collected sediment samples from additional oiled and reference sites (Figure 1, Appendix A). There is limited information regarding the methods that field crews used to collect these sediments; however, any available metadata

Page 1 SC13984

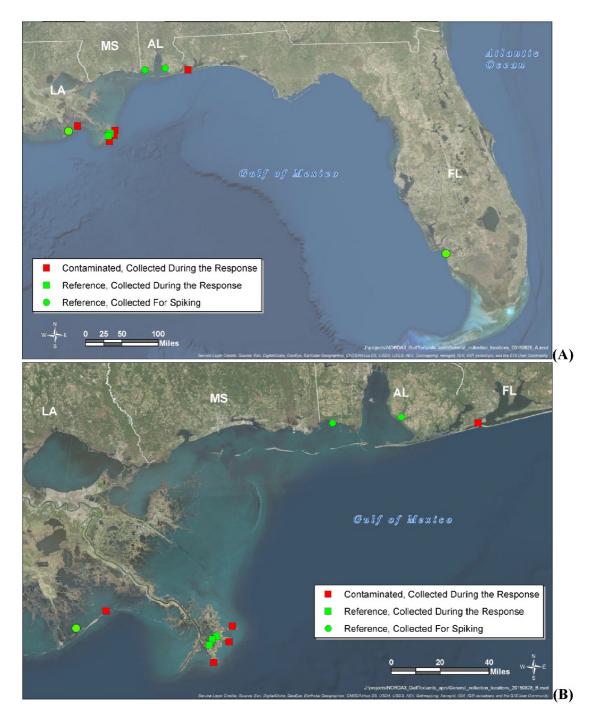


Figure 1. Map of toxicity testing collection sites (A); magnification of map A (B).

Page 2 SC13984

associated with these samples are provided in the National Oceanic and Atmospheric Administration (NOAA) DIVER data repository (DIVER, 2015). After each collection effort, the Trustees shipped the bulk sediment samples to Alpha Analytical in Westborough, MA, for long-term storage. They also sent a small portion of each sample to ALS Environmental (ALS; formerly Columbia Analytical Service) in Kelso, WA or Alpha Analytical for chemical analysis. We classified a site as "contaminated" if it forensically matched with Macondo oil in either 2010 or 2011 (Emsbo-Mattingly and Martin, 2015; Stout and Emsbo-Mattingly, 2015). There are no forensic data for the Black Hole samples; however, we consider these samples contaminated because they were collected in an area that was classified during the NRDA as having "Heavier Persistent" oiling (Nixon et al., 2015).

2.1.2 Sediments collected for spiking

We collected different uncontaminated sediment samples, which we could homogenize with oil, in Alabama, Florida, and Louisiana as a part of the NRDA toxicity program (Figure 1). We classified these samples as uncontaminated because they contained low levels of PAHs (Appendix A). We collected samples in Alabama in 2012 and 2013 using the sampling protocols that are included in Appendix B. In 2012 and 2013 we collected samples in Florida (Estero Bay) using the sampling protocols that are included in Appendix B. In 2013 we collected samples in Louisiana (Caminada Bay), following the work plans included in Appendix C. At the time of the collection, we shipped sediment samples to either Alpha Analytical or ALS, where they were stored at -20°C for long-term storage. In some instances, we shipped the samples directly to the toxicity testing laboratory where they were stored at -20 °C until needed for toxicity testing. We also sent a sample of this sediment to ALS for chemical analysis.

2.2 Analytical Methods

Laboratory personnel at Alpha Analytical (Westborough, MA) or ALS (Kelso, WA) conducted all chemical analyses. They conducted analyses on PAHs, alkyl PAH homologues, and related hetero-compounds using gas chromatography with low-resolution mass spectrometry and selected ion monitoring (GC/MS-SIM), based on U.S. Environmental Protection Agency (EPA) Method 8270D (Morris et al., 2015). We used these data to calculate the sum of 50 PAHs (TPAH50; Forth et al., 2015a).

The analytical laboratory analyzed samples for additional contaminants, including metals (6010C and 6020A) such as antimony (6020A), silver (6020A), and mercury (7471B); pesticides (8081B); and polychlorinated biphenyls (PCBs; 8082A). If possible, personnel also conducted analyses that described the physical properties of the sediments: total organic carbon (TOC; ASTM D4129-05), particle size (PSEP PS), and total solids (TS-MET). Personnel were unable to assess the physical properties of some of the sediment samples because of high oil content.

2.3 Sediment Characterization

2.3.1 Sediments collected during the response

Technicians at the analytical laboratories analyzed samples for PAH, TOC, particle size, and the other contaminants listed above. The chemical and physical properties of these sediments varied. The average properties of all of the sediments collected during the response and used for toxicity testing (reference and contaminated) ranged from approximately 0.09 to 2,170 mg/kg TPAH50 (Figure 2), 0.8% to 55% TOC (Figure 3), and 1% to 94% silt (0.0039–0.0625) + clay (< 0.0039) particle sizes (Figure 4). Appendix A contains the relevant data for these samples.

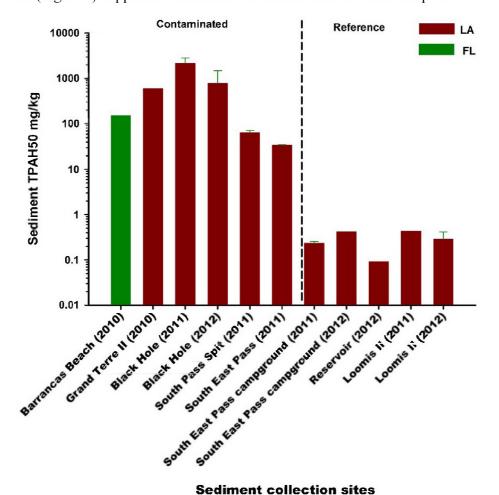


Figure 2. Average TPAH50 concentrations of sediments collected during the response. We calculated these values using the test-specific chemistry at the initiation of a test.

Page 4

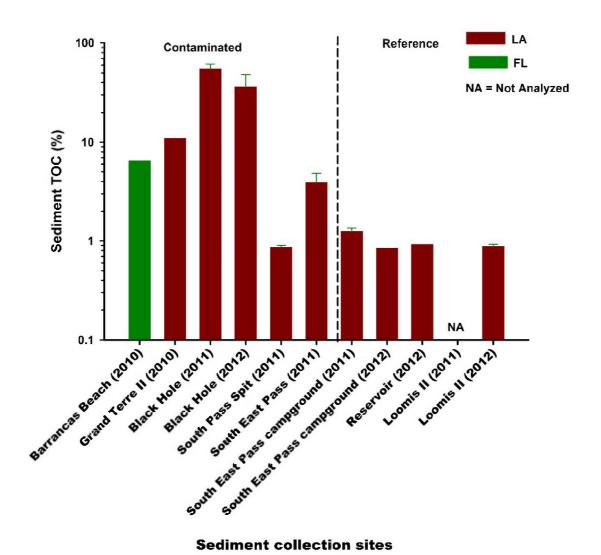


Figure 3. Average TOC values of sediments collected during the response. We calculated these values using the test-specific chemistry at the initiation of a test.

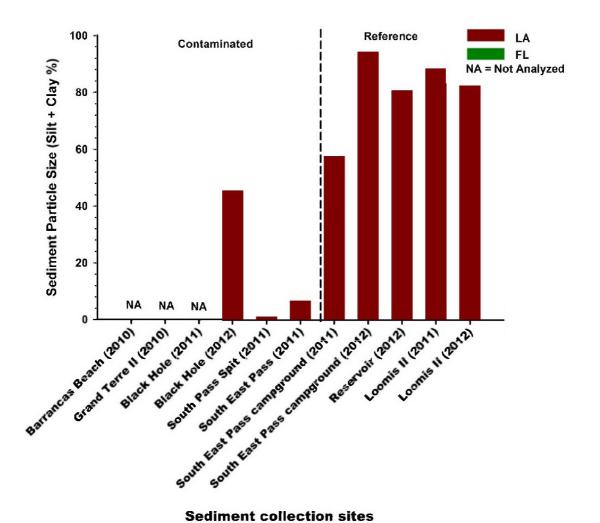


Figure 4. Particle size (silt + clay) of sediments collected during the response.

2.3.2 Sediments collected for spiking

Technicians at the analytical laboratories analyzed samples for PAH, TOC, particle size, and the other contaminants listed above. The chemical and physical properties of these sediments varied. The average properties of all of the sediments collected for spiking ranged from approximately 0.001 to 1.2 mg/kg TPAH50 (Figure 5), 0.2% to -7% TOC (Figure 6), and 0.66% to 99.1% silt (0.0039–0.0625) + clay (< 0.0039) particle sizes (Figure 7). Appendix A contains the relevant data for these samples.

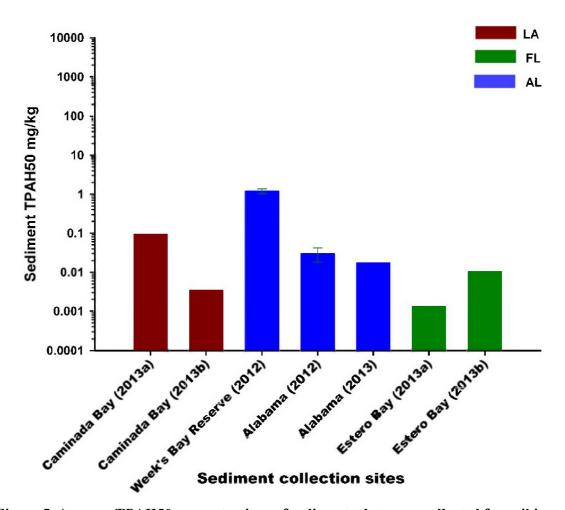


Figure 5. Average TPAH50 concentrations of sediments that were collected for spiking.

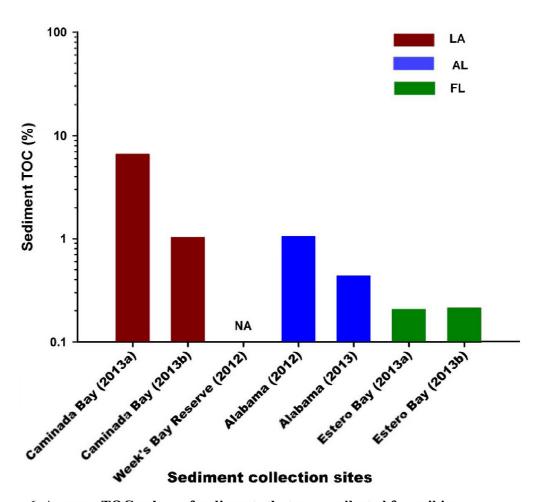
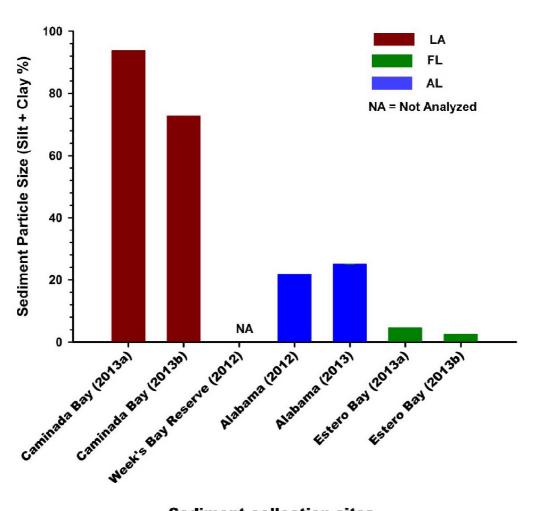


Figure 6. Average TOC values of sediments that were collected for spiking.



Sediment collection sites

Figure 7. Particle size (silt + clay) of sediments that were collected for spiking.

3. Sediment Preparations

During the NRDA sediment toxicity testing program, we conducted bioassays using sediments that were collected during the response effort (Table 1) and sediments that were collected for spiking that were either (1) oiled through the addition of an oil emulsion (i.e., spiked oil emulsion) or (2) homogenized (mixed) with DWH oil (i.e., spiked slick oil) (Table 2).

Table 1. Tests conducted using sediment collected during the response

				Test	ID		
Sediment type	540	569	586	534	576, 609, 610	630	631
Barrancas Beach (2010)						X	
Grand Terre II (2010)						X	
Black Hole (2011)	X	X	X	X	X		X
Black Hole (2012)		X	X	X		X	
South Pass Spit (2011)	X	X	X				X
South East Pass (2011)	X	X	X				X
South East Pass campground (2011)	X	X	X				
South East Pass campground (2012)						X	
Reservoir (2012)						X	
Loomis II (2011)	X						
Loomis II (2012)				X	X	X	

Table 2. Tests conducted using spiked reference sediments

Test ID	Species	Sediment type	Exposure matrix
113	Southern flounder (Paralichthys lethostigma)	Alabama (2012)	Spiked slick oil
114	Amphipod (Leptocheirus plumulosus)	Alabama (2013)	Spiked slick oil
115	Amphipod (Leptocheirus plumulosus)	Alabama (2012)	Spiked slick oil
156	Eastern oyster (Crassostrea virginica)	Estero Bay (2013a)	Spiked slick oil
163	Grass shrimp (Palaemonetes pugio)	Alabama (2013)	Spiked slick oil
195	Gulf killifish (Fundulus grandis)	Alabama (2013)	Spiked slick oil
218	Eastern oyster (Crassostrea virginica)	Estero Bay (2013b)	Spiked slick oil
247	Amphipod (Leptocheirus plumulosus)	Alabama (2013)	Spiked slick oil
256	Eastern oyster (Crassostrea virginica)	Estero Bay (2013b)	Spiked slick oil
262	Amphipod (Leptocheirus plumulosus)	Caminada Bay (2013a)	Spiked slick oil
272	Amphipod (Leptocheirus plumulosus)	Caminada Bay (2013b)	Spiked slick oil
340	Amphipod (Leptocheirus plumulosus)	Caminada Bay (2013b)	Spiked slick oil
342	Amphipod (Leptocheirus plumulosus)	Alabama (2013)	Spiked slick oil
343	Eastern oyster (Crassostrea virginica)	Estero Bay (2013b)	Spiked slick oil
535	Fiddler crab (Uca minax)	Week's Bay (2012)	Spiked oil emulsion
548	Grass shrimp (Palaemonetes pugio)	Alabama (2013)	Spiked slick oil
572	Fiddler crab (Uca longisignalis)	Alabama (2012)	Spiked oil emulsion
578	Fiddler crab (Uca longisignalis)	Alabama (2012)	Spiked oil emulsion

Table 2. Tests conducted using spiked reference sediments (cont.)

Test ID	Species	Sediment type	Exposure matrix
579	Fiddler crab (Uca longisignalis)	Alabama (2012)	Spiked oil emulsion
590	Grass shrimp (Palaemonetes pugio)	Alabama (2013)	Spiked slick oil
634	Southern flounder (Paralichthys lethostigma)	Alabama (2012)	Spiked slick oil
654	Red drum (Sciaenops ocellatus)	Alabama (2013)	Spiked slick oil
655	Pacific white shrimp (Litopenaeus vannamei)	Alabama (2013)	Spiked slick oil
900	Southern flounder (Paralichthys lethostigma)	Alabama (2013)	Spiked slick oil
903	Brown shrimp (Farfantepenaeus aztecus)	Alabama (2013)	Spiked slick oil
904	Southern flounder (Paralichthys lethostigma)	Alabama (2013)	Spiked slick oil
908	White shrimp (Litopenaeus setiferus)	Caminada Bay (2013b)	Spiked slick oil

3.1 Sediments Collected during the Response

During the NRDA sediment toxicity testing program, we conducted tests on southern flounder (540), blue crab (569), grass shrimp (586), eastern oysters (534, 576, 609, and 610), and the amphipod *Leptocheirus plumulosus* (630 and 631) using sediments collected during the response effort (Morris et al., 2015). The testing conditions and protocols between these tests differed. However, each investigator homogenized the sediment and collected samples for PAH analysis before beginning a test (Morris et al., 2015). At the beginning of each test, we collected sediment samples for PAH analysis (Appendix A) and calculated the toxicity of the sediment using the results from these analyses.

3.2 Sediment Oiled with Oil Emulsion

We conducted only a few fiddler crab tests using sediment oiled with an oil emulsion; therefore, we only briefly describe this method here. The purpose of this type of exposure was to simulate an intertidal exposure (i.e., where oil, which was deposited onto the top of the marsh by the tide or waves, slowly seeped into the sediment). To prepare this exposure, we made oil emulsions according to the procedure for preparing high-energy water accommodated fractions (HEWAFs), except that after blending, we used the resultant emulsion in its entirety rather than transferring it to a separatory funnel for a 1-hr separation of layers (Morris et al., 2015). Next, we added the oil:artificial saltwater emulsion to chambers that were filled with reference sediment. After we added the emulsion to these tanks, the tanks underwent a simulated tidal flux to allow the oil to saturate the sediments. A more detailed explanation of the mixing methods can be found in the test-specific protocols (Morris et al., 2015).

3.3 Sediment Spiked with Slick Oil

We homogenized DWH oil and reference sediments to create exposure media for our bioassays. We used four oil types for the broad toxicity testing program: Source oil, Artificially Weathered oil, Slick A oil, and Slick B oil. We conducted sediment toxicity testing using two different oil types: surface slick oil, collected on July 29, 2010, from the hold of barge number CTC02404 (Slick A); and a more weathered surface slick oil, collected on July 19, 2010, by the skimmer vessel USCGC Juniper (Slick B; Forth et al., 2015b; Stout, 2015). We chose Slick A and Slick B oils for sediment testing because these were the most weathered oils (68% and 85% PAH loss, relative to hopane, respectively). We calculated percent depletion of TPAH relative to hopane of all the oils and sediments using the average TPAH and hopane concentrations of over 600 measurements of the fresh Source oil, taken during various Trustee activities, to represent the unweathered oil (Stout, 2015). The 8% PAH depletion for the Source oil shown in Figure 8 is calculated using an analysis of the Source oil used by the DWH NRDA toxicity program compared to this average. Slick B oil was also closer to the average weathering states for the contaminated sediments that we collected for toxicity testing in the field (93%), and the stranded oil samples collected in the field (93%; Stout and Emsbo-Mattingly, 2015; Figure 8). For these reasons, we conducted the majority of our sediment tests using Slick B oil (Morris et al., 2015).

3.3.1 Preparation of spiked sediment

To prepare spiked sediments, we thawed the reference sediment and homogenized it by mixing it at low speed for 2 minutes using a Cuisinart SM-70 7-quart stand mixer or a KitchenAid 5-quart stand mixer. Then we transferred the appropriate amount of oil into the mixing bowl containing the sediment and mixed the oil into the sediment at a medium speed. We stopped the mixer briefly every 2–4 minutes to scrape the sides of the mixing bowl. It took a total of 30 minutes to mix the sediment sample. After the mixing was complete, we either immediately used the samples for toxicity testing or stored the samples at 4°C until they were needed for toxicity testing. A more detailed explanation of the mixing methods can be found in the test-specific protocols (Morris et al., 2015).

Page 12 SC13984

^{1.} Here we describe the weathering state of oil by comparing the total concentration of PAHs in the sample to the concentration of hopane. This is because the total PAH concentration is known to decrease as oil weathers, whereas hopane remains relatively stable because it is not greatly affected by the weathering process (Stout, 2015).

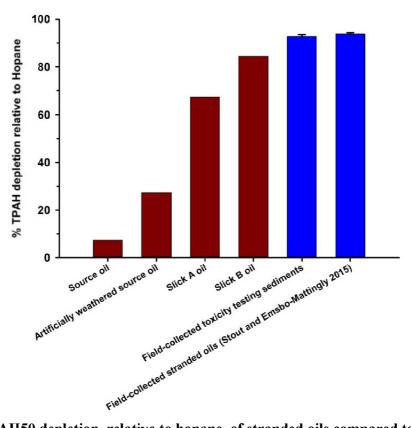


Figure 8. TPAH50 depletion, relative to hopane, of stranded oils compared to field-collected toxicity testing sediments and toxicity oils that could be used to spike sediments for toxicity tests; note that Slick B TPAH50 depletion is closest to that of field-collected samples.

3.3.2 Spiked sediment characterization and analysis

We ran analyses to confirm the homogeneity of our sediment spiking method. To do this, we spiked sediment with $100~\mu g/kg$ of either Slick A or Slick B oil. Then, we collected sediment samples from different quadrants of the mixing bowls that contained the mixture for PAH analysis by ALS. The TPAH50 data from these analyses indicated that our spiking technique produced a fairly homogenous mixture (Figure 9). Although both mixtures were fairly homogenized, we obtained a more homogenous mixture when we spiked sediment with Slick B oil compared to Slick A oil (Figure 9). This was likely due to the different consistencies of the slick oils. The method that we used for spiking the sediment did not alter the composition of the individual PAHs in the oil (Brown-Peterson et al., 2015).

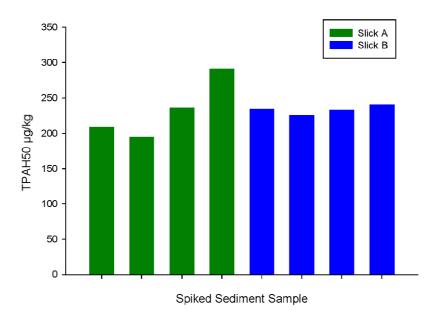


Figure 9. TPAH50 values for sediment samples from different quadrants of a bowl mixed with either slick oil.

4. Discussion

During the DWH NRDA toxicity testing program, we used a variety of sediment exposure matrices. We used sediments that came directly from areas affected by the spill to simulate field conditions. This type of exposure allowed us to directly test the toxic effects of sediments that were contaminated with DWH oil. However, the sediments differed from one another in their physical properties, such as particle size and TOC. The physical properties of sediment may affect the toxicity (U.S. EPA, 2003); therefore, we conducted additional toxicity tests using sediment that controlled for these differences. The EPA Equilibrium Partitioning Sediment Benchmarks for PAH mixtures reports that TOC can affect toxicity by reducing the bioavailability of PAHs in interstitial water (i.e., sediment pore water; U.S. EPA, 2003). By spiking a single type of sediment with different concentrations of oil, we were able to maintain consistent particle size distributions and TOC concentrations during each bioassay. While we are aware of the potential effects of TOC on toxicity, EPA partitioning benchmarks only consider dissolved PAHs in interstitial water (U.S. EPA, 2003); other PAHs that adsorb to the sediment are also toxic (Morris et al., 2015), so only considering the dissolved fraction may underestimate toxicity.

We conducted toxicity tests with both field-collected contaminated sediments that varied in DWH oil concentration, TOC, and particle size, and sediments that were spiked with varying levels of DWH oils. Morris et al. (2015) provide details regarding the methodology and results of the different sediment tests that were conducted during the DWH NRDA.

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A. Supporting Chemistry

Table A.1. Supporting information for the sediments collected during the repossess effort

Sample ID	Sediment type	Latitude	Longitude	Fines (%)	Forensic match
LAAR38-B0125-SX404	Black Hole (2011)	29.19689	-89.039	N/A	N/A
LAAR38-B0125-SX405	South Pass Spit (2011)	28.98487	-89.1717	1.03	Yes
LAAR38-B0125-SX410	Loomis II (2011)	29.12305	-89.1787	88.32	No
LAAR38-B0126-SX406	South East Pass (2011)	29.10449	-89.0645	6.69	Yes
LAAR38-B0126-SX412	South East Pass Campground (2011)	29.13911	-89.1486	57.67	No
LAAR42-C0208-SX404	Loomis II (2012)	29.12305	-89.1787	82.38	N/A
LAAR42-C0208-SX406	Reservoir (2012)	29.08977	-89.1984	80.65	N/A
LAAR43-C0208-SX402	South East Pass Campground (2012)	29.13911	-89.1486	94.26	N/A
OAAQ39-0-10-A0822-S6510	Grand Terre II (2010)	29.3131	-89.8998	N/A	Yes
OAAQ39-0-1-A0822-S6501	Grand Terre II (2010)	29.30697	-89.8706	N/A	Yes
OAAQ39-0-5-A0822-S6505	Grand Terre II (2010)	29.30615	-89.8867	N/A	Yes
fg-c0926-se-534-204	Black Hole (2012)	29.19689	-89.039	45.43	N/A
FLAK50-A0924-B116004	Barrancas Beach	30.34026	-87.3136	N/A	Yes

Table A.2. TPAH50 and TOC data from several toxicity tests for the sediments collected during the response effort

Sample ID	Sediment type	тос	TPAH50 mg/kg ND(0)
pe-d0129-se-630-102	Barranca's Beach (2010)	6.46	150
um-c0721-se-569-105	Black Hole (2011)	58.8	2,086
gr-c0906-se-586-105	Black Hole (2011)	37.8	31
gr-c0502-se-540-102	Black Hole (2011)	N/A	3,940
fg-c0926-se-534-203	Black Hole (2011)	69.9	3,259
pe-d0301-se-631-102	Black Hole (2011)	52.7	1,532
pe-d0129-se-630-106	Black Hole (2012)	15.1	109
um-c0721-se-569-106	Black Hole (2012)	31.9	39
gr-c0906-se-586-106	Black Hole (2012)	70.6	2,880
fg-c0926-se-534-204	Black Hole (2012)	26.5	61
pe-d0129-se-630-101	Grand Terre II (2010)	10.9	602
gr-c0502-se-540-104	Loomis II (2011)	N/A	0
pe-d0129-se-630-104	Loomis II (2012)	0.926	0
fg-c0926-se-534-201	Loomis II (2012)	0.833	0
pe-d0129-se-630-105	Reservoir (2012)	0.92	0
um-c0720-se-569-102	South East Pass (2011)	2.37	36
gr-c0906-se-586-103	South East Pass (2011)	3.63	36
gr-c0502-se-540-103	South East Pass (2011)	N/A	33
pe-d0301-se-631-104	South East Pass (2011)	5.68	32
um-c0720-se-569-101	South East Pass campground (2011)	1.14	0
gr-c0906-se-586-101	South East Pass campground (2011)	1.35	0
gr-c0502-se-540-105	South East Pass campground (2011)	N/A	0
pe-d0129-se-630-103	South East Pass campground (2012)	0.848	0
um-c0721-se-569-104	South Pass Spit (2011)	0.939	70
gr-c0906-se-586-104	South Pass Spit (2011)	0.85	55
gr-c0502-se-540-101	South Pass Spit (2011)	N/A	81
pe-d0301-se-631-103	South Pass Spit (2011)	0.796	47

Table A.3. Supporting information for the sediments collected for spiking

Sample ID	Site name	Latitude	Longitude	TOC (%)	TPAH50 mg/kg ND(0)	Fines (%)
AU-C0625-SE-535-153	Alabama (2012)	30.37937	-88.30678	1.05	0.042	21.73
AU-C0625-SE-535-154	Alabama (2012)	30.37937	-88.30678	1.05	0.018	21.77
ALAJ46-D0515-S34501	Alabama (2013)	30.37937	-88.30678	0.434	0.018	24.97
gr-e0122-se-g08-101	Caminada Bay (2013a)	29.21101	-90.09805	6.6	0.094	93.81
LAAQ38-D1203-CAM	Caminada Bay (2013b)	29.21101	-90.09805	1.03	0.003	72.85
FLBH74-D0531-S34901	Estero Bay (2013a)	26.35018	-81.84217	0.205	0.001	4.51
FG-D0920-SE-218-900	Estero Bay (2013b)	26.35018	-81.84217	0.212	0.011	2.49
AU-C0515-SE-535-125	WeeksBay	30.396272	-87.834374	N/A	1.061	N/A
AU-C0515-SE-535-126	Weeks Bay	30.396272	-87.834374	N/A	1.366	N/A

B. Sampling Protocols Used for 2012 and 2013 Alabama and Florida Collections

Sediment Collection Instructions

Sediment Collection:

- 1. Line a 5 gallon bucket with two heavy duty garbage bags.
- 2. Using a shovel, collect sediment from the top 6-8 inches, avoiding obvious large debris such as rocks, sticks, vegetation, etc., and place sediment in 5 gallon bucket double-lined with heavy duty garbage bags.
- 3. Continue collecting sediment until 5 gallon bucket is full.
- 4. If sediments contain significant water, allow collected sediment to rest for a 5-10 minutes to let sediment settle, and then remove accumulated water on top of sediments. After water is decanted, top off bucket with additional sediments, so that a full 5 gallons of sediments are in each bag collected.
- 5. Once the 5 gallon bucket is filled, using a small scoop or shovel, grab a small aliquot of sediment, and transfer to a large metal bowl. This will be used in a composite sediment sample that is collected from each of the 14 x 5 gallon bags of sediment. This sample will be sent to ALS Environmental for analysis (COC also provided by Stratus).
- 6. After sediment aliquot is taken, close up garbage bags using a zip tie.
- 7. Label bag by folding a piece of tape around bag closure and back on itself. Label should contain the Sample ID and bag number (e.g., 1 of 14).
- 8. Transfer full garbage bag of sediments from 5 gallon bucket to a cooler for shipment to Alpha Analytical. See below for packaging and shipment instructions.
- 9. Continue until 14 x 5 gallon bags of sediment are collected.
- 10. Transfer composited sediments to four 8 oz sediment sample jars for shipment to ALS Environmental.

Packaging and Shipment of 5 gallon bags:

- 1. Each cooler should contain one 5 gallon bag of sediment.
- 2. Chain of Custody (COC) forms will be provided by Stratus Consulting (one for each cooler) in individual plastic resealable bags. The forms will have the shipper information

and most of the sample information filled out. There is a section of the sample ID that requires the date of sampling. The month and day will be left blank, to be filled out by the sample collector(s) with the correct month and day of the sampling date. See example sample ID below, where MM is the month and DD is the day of sampling.

ALAJ46-FMMDD-S34501

- 3. In addition, the sample date and time should be filled out in the adjacent cells.
- 4. After the Sample ID, date, and time have been competed on each COC form, the sample collector(s) should sign and date the bottom of the form under the "Relinquished By" section
- 5. Place completed COC forms into the plastic resealable bags that they came in and attach to garbage bags with sediment.
- 6. To attach COC forms to garbage bags with sediment run a zip tie through the hole punched at the top of the sealed plastic bag with COC form and then cinch the zip tie with COC form around top of the garbage bags.
- 7. Sign and date at least two COC seals (small stickers provided by ALS Environmental with a line for a signature) for each shipping container.
- 8. Place signed COC seals on opposite corners of the cooler across the seam between the cooler lid and the main body of the cooler (Figure 2). COC seals must be arranged so that the cooler cannot be opened without disturbing the seals.
- 9. Place clear packing tape over the COC seals.
- 10. Seal the cooler by taping around the seam between the lid and body of the cooler and around the entire cooler (Figure 3).
- 11. Deliver cooler(s) to a FedEx location or have FedEx pick up the cooler(s). **Do not** leave the cooler(s) at an unattended FedEx drop-off location. Samples will be shipped using standard overnight delivery as soon as possible following collection. Avoid shipping samples over weekends. The FedEx code to use is

All 5 gallon bags of sediment should be shipped to Alpha Analytical. Their shipping address is:

Alpha Analytical 320 Forbes Boulevard

Mansfield, MA 02048

Attn: Wendy Wong/Susan O'Neil

For the analytical sample jars, these are to be shipped to ALS Environmental in Kelso, Washington, using our standard shipping protocols described in the QAPP. The shipping address for ALS Environmental is:

ALS Environmental 1317 South 13th Avenue Kelso, WA 98626 800-695-7222 (telephone)

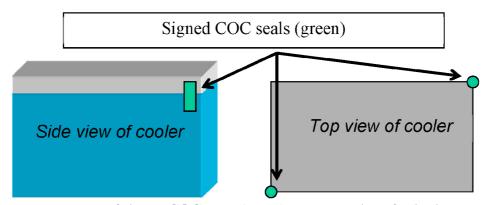


Figure 2. Placement of signed COC seals (green) on the outside of shipping coolers.

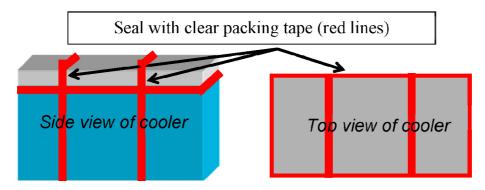


Figure 3. Placement of clear packing tape (red lines) on shipping coolers.

C. Work Plans for the Caminada Bay Sediment Collection Efforts

LOSCO – Task 26 Caminada Bay Sediment Sampling Documentation

5 August 2013 Michael Carney, Stratus Consulting

Contents:

- 1. Work plan (2 pages)
- 2. Field Datasheets (5 pages)
- 3. Copies of original Chain of Custody forms (18 pages)
- 4. Copies of GCRL Binnaz Baily received Chain of Custody forms (5 pages)

Caminada Bay Sediment Sampling

1. Introduction and Purpose

This work plan describes the proposed field sampling of surficial marine sediment from Caminada Bay near Grand Isle, Louisiana.

1.1 Study Objective

The primary study objective is to collect, distribute, and store sediment from the Caminada Bay area for toxicity testing related to the *Deepwater Horizon* NRDA

The primary study objective will be met through the following specific activities:

- Collect approximately 75 gallons of surficial sediments from around one location in Caminada Bay, LA.
- Maintain an inventory of sediment collections detailing the volume, location, and use of each aliquot; including monitoring the volumes necessary to adhere to sample retention requirements.
- Transport approximately half of collected sediments to Gulf Coast Research Laboratory (GCRL), Ocean Springs, MS and store according to strict chain of custody (COC) requirements.
- Ship approximately half of collected sediments to ALS Environmental, Kelso, WA under strict COC requirements.

2. Sampling and Analysis Approach and Methods

The proposed sampling approach is to collect sediment samples at a predetermined sampling location within Caminada Bay near Grand Isle, Louisiana. We anticipate collecting sediment on August 5, 2013. The actual sampling location will be selected in the field. The Field Team Leader may move the sample location, identify additional sampling locations, or exclude proposed sampling locations, based on field conditions and observations. Factors influencing sample numbers and locations may include weather conditions such as high winds that make certain sample locations unsafe, sediment substrate composition such has hard bottom that

Page 1
DWH ATTORNEY WORK PRODUCT/ATTORNEY-CLIENT COMMUNICATIONS

sampler cannot penetrate, or the presence of petroleum sheen or odor that suggests potential sediment contamination.

Sediments will be collected using a newly decontaminated petite PONAR sampler or similar apparatus from approximately the top 10 cm of the sediment column. The sub-samples (each PONAR grab) will be placed in a cooler lined with new heavy duty plastic bags; alternating coolers for each sub-sample. This process will be repeated until the required volume of sediment is collected. Approximately, 75-gallons of sediment will be collected.

Samples will be collected at the predetermined global positioning system (GPS) coordinates (29.21101, -90.09805). They will be taken in an up-current progression, beginning with the locations that are most down-current of the tidal flow. If sampling begins during a period of no tidal movement, it will progress from the down-current side of the impending tidal flow to the up-current sampling locations. The boat will be anchored with the motor off and out of the water when sediments are being collected.

Sediment will be processed on board the sampling vessel immediately following collection at each location. Sediment will be visually inspected for signs of contamination. Any such signs will be documented via photographic and field notes; after the sample is collected, any leftover portion of apparently contaminated sediment will be containerized on board the vessel for proper disposal by the State at an approved facility. All sediments collected will be packaged on ice and personally delivered to Dr. Joe Griffitt at the GCRL in Ocean Springs, MS or shipped under chain of custody (COC) to ALS Environmental in Kelso, WA.

3. Project Staff

We recommend that individuals experienced in subaqueous sediment collection and inspection perform these sampling efforts. These individuals may be available through appropriate State agencies (e.g., Department of Wildlife and Fisheries and/or Department of Natural Resources) or LOSCO may be accessible through existing State contractors. Mr. Michael Carney of Stratus Consulting will oversee this sampling effort and serve as Field Team Leader.

Page 2
DWH ATTORNEY WORK PRODUCT/ATTORNEY-CLIENT COMMUNICATIONS

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Contact/Phone/Email: Michal Affiliation:	uel Carn			4		Contact	Phone/En	nail:	Michael Carney 303-381-8000 (office) or 919 mcarney@stratusconsulting. or Ryan Takeshita 303-381-8000 (office) or 505 rtakeshita@stratusconsulting	-690-5527 (cell)
Project Name:	LOSCO Cam	inada Bay S	ediment Collec	tion						Storage or destination lab cont⊬ct information
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Contact/Phone/Email: Mic/ Affiliation: 5 to 1244.5	Mael Carr	hey				Michael Carney 303-381-8000 (office) or 919-452-1861 (cell) mcarney@stratusconsulting.com or Ryan Takeshita 303-381-8000 (office) or 505-690-5527 (cell) rtakeshita@stratusconsulting.com					
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Stratus	s Consu	149								3000 (office) or 50 @stratusconsulti	05-690-5527 (cell) ng.com	
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	Sampler/Sl	nipper Inforn	nation						₽ro	ject Cont	act Infor	mation
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Cammada LOC # 04



Sample/Shipper Information Contact/Phone/Email: Michael Carney 303-381-8000 (office) or 919-452-1861 (cell) macriney@stratusconsulting.com or Ryan Takeshita 303-381-8000 (office) or 505-690-5527 (cell) rakeshita@stratusconsulting.com Storage or destination lab contact information Storage or destination lab contac	nples, data, or any atory analyses COC Form of
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Work Plan and Field Data for Round 2 of LOSCO Task Order 26 - Caminada Bay Sediment Collection

Contents:

- ▶ Field work plan: 2 pages
- Field datasheets: 5 pages
- Sediment COC forms: 20 pages for 5-gallon lots shipped to FAU and ALS; 1 page for 32-oz sent to ALS for chemical analysis COCs total 21 pages
- Sediment lot Southwest Cargo shipping airbills: 2 pages

Caminada Bay Sediment Sampling

1. Introduction and Purpose

This work plan describes the proposed field sampling of surficial marine sediment from Caminada Bay near Grand Isle, Louisiana.

1.1 Study Objective

The primary study objective is to collect, distribute, and store sediment from the Caminada Bay area for toxicity testing related to the *Deepwater Horizon* NRDA

The primary study objective will be met through the following specific activities:

- Collect approximately 75 gallons of surficial sediments from around one location in Caminada Bay, LA.
- Maintain an inventory of sediment collections detailing the volume, location, and use of each aliquot; including monitoring the volumes necessary to adhere to sample retention requirements.
- Ship approximately half of collected sediments to Florida Atlantic University, Boca Raton, FL and store according to strict chain of custody (COC) requirements.
- Ship approximately half of collected sediments to ALS Environmental, Kelso, WA under strict COC requirements.

2. Sampling and Analysis Approach and Methods

The proposed sampling approach is to collect sediment samples at a predetermined sampling location within Caminada Bay near Grand Isle, Louisiana. We anticipate collecting sediment on December 3, 2013. The actual sampling location will be selected in the field. The Field Team Leader may move the sample location, identify additional sampling locations, or exclude proposed sampling locations, based on field conditions and observations. Factors influencing sample numbers and locations may include weather conditions such as high winds that make certain sample locations unsafe, sediment substrate composition such has hard bottom that sampler cannot penetrate, or the presence of petroleum sheen or odor that suggests potential sediment contamination.

Page 1
DWH ATTORNEY WORK PRODUCT/ATTORNEY-CLIENT COMMUNICATIONS

Stratus Consulting (11/20/2013)

Sediments will be collected using a newly decontaminated petite PONAR sampler or similar apparatus from approximately the top 10 cm of the sediment column. The sub-samples (each PONAR grab) will be placed in a cooler or 5-gallon bucket lined with new heavy duty plastic bags; alternating coolers/buckets for each sub-sample. This process will be repeated until the required volume of sediment is collected. Approximately, 75-gallons of sediment will be collected.

Samples will be collected at the predetermined global positioning system (GPS) coordinates (29.21058, -90.09810). They will be taken in an up-current progression, beginning with the locations that are most down-current of the tidal flow. If sampling begins during a period of no tidal movement, it will progress from the down-current side of the impending tidal flow to the up-current sampling locations. The boat will be anchored with the motor off and out of the water when sediments are being collected.

Sediment will be processed on board the sampling vessel immediately following collection at each location. Sediment will be visually inspected for signs of contamination. Any such signs will be documented via photographic and field notes; after the sample is collected, any leftover portion of apparently contaminated sediment will be containerized on board the vessel for proper disposal by the State at an approved facility. All sediments collected will be packaged on ice in the field. Gel ice packs will be used when sediments are prepared for shipping. Sediments will be shipped using a commercial airport to airport carrier under COC.

3. Project Staff

We recommend that individuals experienced in subaqueous sediment collection and inspection perform these sampling efforts. These individuals may be available through appropriate State agencies (e.g., Department of Wildlife and Fisheries and/or Department of Natural Resources) or LOSCO may be accessible through existing State contractors. Mr. Michael Carney of Stratus Consulting will oversee this sampling effort and serve as Field Team Leader.

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Sample Shipment contains USDA regulated soil samples (check box if applicable)

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